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AZUSA PLANT

STRUCTURAL MATERIALS DIVISION

INVESTIGATION OF STRESS-CORROSION CRACKING
OF HIGH-STRENGTH ALLOYS

A Report To

FRANKFORD ARSENAL

Contract DA-04-495-ORD-3069

Report No. L0414-01-21 / April 1963 / Copy No.

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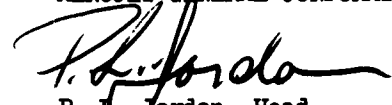
ASTIA
APR 22 1963



This is the twenty-first in a series of informal monthly progress reports submitted in partial fulfillment of Contract DA-04-495-ORD-3069. It constitutes the fifth monthly progress report for the one-year continuation of the original two-year program.

This report covers the period 1 January through 31 January 1963. It was written by R. B. Setterlund who was supervised by A. Rubin.

AEROJET-GENERAL CORPORATION



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NOTE: The information contained herein is regarded as preliminary and subject to further checking, verification, and analysis.

I. OBJECTIVES

The objectives of this program are outlined below:

A. Investigation of the stress-corrosion cracking characteristics of at least three new high strength alloys of interest for rocket motor case applications. These alloys are 6Al-4V titanium, 18%-nickel maraging steel, and 20%-nickel maraging steel, in addition to limited testing of vacuum-melted 9Ni-4Co steel.

B. Study of the environmental parameters that could affect the rate and extent of stress-corrosion cracking.

C. Determination of the effect of material parameters (composition, strength level, welding, and microstructure) on stress-corrosion susceptibility.

D. Continuation of the evaluation of protective coatings and other techniques for preventing stress-corrosion cracking.

II. SUMMARY

Data obtained to date show that the 6Al-4V titanium alloy is immune to stress-corrosion cracking under all the test conditions of this program in both the annealed and in the quenched-and-aged conditions.

The 20%-nickel maraging steel was found to be susceptible to stress corrosion cracking in both the annealed-and-aged and in the 75% cold-worked conditions. The susceptibility is much greater, however, in the annealed condition.

The 18%-nickel maraging steel was also found to be susceptible to stress corrosion cracking in both the annealed-and-aged and cold-worked-and-aged conditions. As with the 20%-nickel alloy the annealed and aged condition showed the greater susceptibility.

Among the test environments employed, the most severe exposures were those in moisture-saturated air at 140°F, a 3% salt water solution, and distilled water.

Fifteen different coating systems designed to prevent stress-corrosion cracking are under evaluation using the failure-susceptible steel, H-11, as the base material. Several inhibited epoxy systems show a promising capability for protecting the metal.

III. WORK PROGRESS

A. INTRODUCTION

Since the initiation of the original test program two years ago, to investigate the stress-corrosion cracking characteristics of high-strength alloys, a number of new high-strength steels have been receiving increased attention for use in constructing rocket motor cases. The third year test program is directed to the study of three of these new alloys as well as of one titanium alloy presently being used for the same application.

The test environments, substantially the same as those evaluated in the original two-year investigation, are as follows: (1) distilled water; (2) tap water; (3) salt water; (4) sodium dichromate-inhibited water; (5) soluble oil-inhibited water; (6) air; (7) high humidity atmosphere; (8) trichloroethylene; (9) cosmoline; and (10) solid propellant. These are considered to be environments representative of those to which rocket motor cases would normally be exposed during fabrication, processing, or storage. One additional environment will be included in the new program, that of sea-coast exposure.

The test methods being used in this investigation employ bent-beam, U-bend, and center-notch specimens. Evaluation of results includes microstructural studies, using both standard metallographic and electron microscopic techniques, to attempt to associate the failure mechanism with specific microstructural characteristics of the materials.

An evaluation of protective coatings and surface treatments to prevent stress-corrosion cracking is also being conducted.

B. PROGRAM STATUS

Bent beam specimens of the 6Al-4V alloy were removed from test after 1700 hours of exposure to the various environments in this program. The material had been processed as shown in Table 1. Examination of these samples indicated no evidence of cracking. Likewise no failures were obtained in 100-hour testing of center-notched specimens. Welded plates have now been fabricated and X-rayed for joint integrity, and specimens are now being prepared for evaluation of the remaining test condition, welded joints.

The 20%-nickel maraging steel is under test in the annealed-and-aged and 75% cold-worked-and-aged conditions (Code H-1 and H-3, Table 1). Specimens are now being machined for testing of the 50% cold-worked-and-aged material (H-2). Center-notch specimen tests are almost complete with the H-1 and H-3 material. A portion of the heat of H-1 material is now being welded using a TIG process.

The 18%-nickel maraging steel is under test with the annealed-and-aged (I-1) and 50% cold-worked-and-aged (I-3) material having a titanium content of 0.62%.

Annealed and cold-worked heats of this same alloy with a titanium content of 0.50% have just been received and will shortly be in test. The determination of the effect of titanium content on stress-corrosion cracking (another objective of this program) is being conducted with limited quantities of material from another program. The chemical analyses of these heats (Nos. 477, 448, and 476, shown in Table 2) indicate a titanium content varying from 0.40 to 1.00%. Welding is soon to be started on the annealed 0.50% titanium heat.

The 9Ni-4Co vacuum-cast alloy is now scheduled for delivery in March. Shipment delays were caused by difficulties at the mill in producing a satisfactory heat.

C. TEST RESULTS TO DATE

The 6Al-4V titanium alloy was completely immune to stress corrosion cracking in any of the test environments of this program. The immunity was

indicated in the bent-beam tests as well as in the more sensitive center-notched tensile tests which were performed, and this was true for both the annealed and the quenched-and-aged processing conditions. Tables 3 and 4 present, respectively, the chemical analysis and mechanical properties, and the effects of stress corrosion, of 6Al-4V alloy.

Test results to date involving bent-beam and center-notch specimens of the maraging steels are shown in Table 5. Both the 20% and 18% maraging steels in both the annealed-and-aged and cold-worked-and-aged conditions showed some failures in these tests. However, there was a wide variation in susceptibility to cracking in different environments. For example, the annealed-and-aged 20%-nickel steel failed more rapidly in distilled water and salt water than the annealed-and-aged 18%-nickel grade; yet the latter material failed in tap water, chromate solution, and soluble oil solutions, while the annealed-and-aged 20%-nickel steel was immune to cracking in these environments. When these same alloys were cold-worked before aging their resistance to cracking was greatly increased. Also the mode of cracking appeared to change from intergranular to possible cracking along slip planes. Photomicrographs of both types of failures were included in the previous formal quarterly report (0414-01-8).

The results of the coating evaluation program are shown in Table 6. Fifteen different coatings are under test in both an aerated salt water environment and a high humidity atmosphere. Several of the coatings appear to show promise for protecting a highly susceptible alloy such as H-11 steel (used in these tests) from stress corrosion cracking. The inhibited epoxy coatings appear to be very effective in protecting the metal from cracking but longer exposures will be needed before any final conclusions can be drawn. Although the vinyl coating is the only type that has not failed in either the salt solution or high humidity tests, the exposure time is still relatively short.

IV. FUTURE WORK

Work will continue along the guidelines of the Master Plan shown in Table I. Both bent-beam and center-notched specimens will be immersed as required to fulfill

this schedule. It is hoped that the 9 Ni-4Co vacuum-cast alloy will be shipped within the next few weeks as promised by the supplier, so that some limited testing of this alloy can be started. Exposure of the maraging steels to the sea coast atmosphere is ready to begin; some results will be shown in the next monthly report.

Metallographic sections of selected cracked samples have been prepared and photographed. In addition, the cracking process is being studied by means of the electron microscope, utilizing fracture replicas. The intention is to attempt to define the mode of failure and, if possible, associate the failure process with microstructural characteristics of the materials. Both photomicrographs and electron microscope fractographs will be presented in the next quarterly report.

V. BUDGET

The expenditure rate for the month of January was 480 hours, leaving a total of 1200 hours to be expended on the remainder of the program.

TABLE 1
MASTER PLAN - BENT BEAM STRESS-CORROSION TESTS

Alloy	Processing Condition (Titanium Content of Welding Steel Shown)	Strength Level, 0.2% Offset Yield (psi)	Specimen Code	Number of Test Environments										See Coast Atmosphere	Total
				Distilled Water	Tap Water	3% NaCl Solution	0.2% Sodium Dichromate Solution	1% Soluble Oil Solution	High Humidity	Trichloro- ethylene	Comsoline	Solid Propellant	Ambient Air		
Gal 4V titanium	Annealed	125,000	G-1	3	3	3	3	3	3	3	3	3	3	3	30
	Quenched and Aged	165,000	G-2	3	3	3	3	3	3	3	3	3	3	3	30
	Welded	135,000	G-W	3	3	3	3	3	3	3	3	3	3	3	30
	Total			8	8	8	8	8	8	8	8	8	8	8	80
20% Nickel Welding Steel	Annealed and Aged	291,000	H-1	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW and Aged	321,000	H-2	3	3	3	3	3	3	3	3	3	3	3	33
	75% CW and Aged	298,300	H-3	3	3	3	3	3	3	3	3	3	3	3	33
	Welded and Aged	To be tested	H-W	3	3	3	3	3	3	3	3	3	3	3	33
	Total			12	12	12	12	12	12	12	12	12	12	12	132
10% Nickel Welding Steel	Annealed & Aged (0.65% Ti)	283,000	I-1	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.50% Ti)	302,400	I-2	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.65% Ti)	323,000	I-3	3	3	3	3	3	3	3	3	3	3	3	33
	Welded & Aged (0.50% Ti)	249,900	I-4	3	3	3	3	3	3	3	3	3	3	3	33
10% Nickel Welding Steel	Annealed & Aged (0.40% Ti)	278,000	I-5	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.50% Ti)	275,400	I-6	3	3	3	3	3	3	3	3	3	3	3	33
	50% CW & Aged (0.55% Ti)	331,000	I-7	3	3	3	3	3	3	3	3	3	3	3	33
	Welded & Aged (1.00% Ti)	323,200	I-8	3	3	3	3	3	3	3	3	3	3	3	33
9 Ni-4 Co Vacuum- Cast Alloy	Annealed & Aged (1.00% Ti)	354,400	I-9	3	3	3	3	3	3	3	3	3	3	3	33
	Welded & Aged (0.50% Ti)	To be tested	I-W	3	3	3	3	3	3	3	3	3	3	3	33
	Total			30	15	23	15	15	30	15	15	15	25	27	225
	Aged (0.25-0.30% C)	To be tested	J-1	3	3	3	3	3	3	3	3	3	3	3	33
H-11 Steel (Coating Tests)	Aged (0.40-0.45% C)	To be tested	J-2	3	3	3	3	3	3	3	3	3	3	3	33
	Application of Various Protective Coatings			6	6	6	6	6	6	6	6	6	6	6	66
	Total			9	9	9	9	9	9	9	9	9	9	9	99
				56	41	86	41	41	85	41	41	39	43	26	395

* Number of replicate tests conducted.

Table 1

TABLE 2
CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES OF MANGING STEELS

Mill-Certified Analysis (Percent Composition)																
Supplier	Heat Numbers	C	Mn	P	S	Si	Ni	Co	Mo	Al	Cr	Zr	Ti	Ca	B	
Allegheny Inadium	W-24254	0.009	0.09	0.002	0.005	0.06	20.41	-	4.92	0.29	0.39	0.002	1.40	0.004	0.003	
	W-24178	0.012	0.01	0.003	0.005	0.01	18.69	8.90	-	0.029	-	0.005	0.62	0.006	0.002	
	477	0.018	0.002	0.006	0.004	0.024	18.29	9.10	4.95	0.089	-	<0.004	0.40	<0.0006	<0.003	
	448	0.029	0.002	0.004	0.008	0.009	18.51	8.46	4.92	0.089	-	<0.004	0.52	<0.0006	<0.003	
	476	0.020	0.002	0.006	0.005	0.014	18.60	9.05	4.90	0.078	-	<0.004	1.00	<0.0006	<0.003	
Republic	2960902	0.020	0.08	0.007	0.006	0.15	18.48	7.00	4.84	0.21	-	0.035	0.50	Added	.0036	

Mechanical Properties (Aerojet Tests)										
Supplier	Heat Numbers	Percent Cold Reduction	Aging Treatment	Table 1		Table 2		Table 3		Hardness (Rockwell C)
				Code No.	0.2% Offset Y.S. (psi) (Transverse)	U.T.S. (psi) (Transverse)	Percent Elongation (in 2 in.)	Percent Reduction In Area	Notched Tensile Strength (psi)	
W-24254		0	None		128,500	170,700	7.5	53	-	34
		0	-100°F + 850°F 4 hr	H-1	291,500	302,200	3	17	58,200	54
		50	None		184,000	201,600	5	50	-	39
		50	850°F 4 hr	H-2	321,000	327,100	3	25	-	55.5
		75	None		205,700	220,900	6	46	-	55
W-24178		75	850°F 4 hr	H-3	298,500	308,800	2.5	13	31,500	55
		0**	None		102,000	153,300	14	62	-	30.5
		0**	900°F 3 hr	I-1	285,000	294,000	8	38	179,100	53.5
		50	None		167,700	189,000	3.5	51	-	36.5
		50	900°F 3 hr	I-3	323,800	368,400	1.5	28	113,500	55
477		50	None		169,500	196,900	6.5	40	-	38.5
		50	900°F 3 hr	I-5	278,000	280,700	2	8	158,800	55
448		0	None		105,500	150,500	10	45	-	30.5
		0	900°F 3 hr	I-6	255,400	265,900	5	9	191,600	52
476		50	None		175,500	199,800	4.5	47.5	-	38
		50	900°F 3 hr	I-7	331,000	332,500	1.5	7	175,000	55
		0	None		128,500	174,700	5.5	48	-	36
		0	900°F 3 hr	I-8	325,500	350,000	2.5	27	162,000	56
		50	None		192,200	217,000	2.5	40	-	41
	50	900°F 3 hr	I-9	354,400	354,900	1	1.5	95,400	58	

TABLE 3CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES
OF 6Al-4V TITANIUM

	Chemical Analysis (% Composition)*								
	C	Al	V	O ₂	N ₂	H ₂	Ti	Fe	Other
Aerojet Analysis	0.3	6.1	4.1	0.083	0.014	80 ppm	Bal	0.16	0.18

	Mechanical Properties (Transverse)			
	Yield Strength Ultimate (0.2% Offset) (psi)	Strength (psi)	Elongation (%)	Hardness (R _c)
Annealed				
Mill report	131,900	141,400	12	33.5
Aerojet test	138,000	143,800	14	34
Notched tensile strength**	--	128,500	-	-
1675°F 1 hr, W.Q. Aged 900°F 8 hr				
Aerojet test	162,700	176,800	7	38.5
Notched tensile strength	--	132,000	-	-
Welded				
Aerojet test	131,500***	135,200	9.5	33.0

* Titanium Metals Corporation HT 4141.

** Using as-fatigue-cracked sample of Figure 3.

*** Tensile failures in parent metal.

TABLE 4

STRESS CORROSION OF 6Al-4V TITANIUM
IN VARIOUS ENVIRONMENTS

Environment	Condition G-1*			Condition G-2		
	Failed/Tested	Failure Times		Failed/Tested	Failure Times	
		Mean (hr)	Range (hr)		Mean (hr)	Range (hr)
Annealed (as received)						
1675° F 1 hr, W.Q., 900° F 8 hr						
Bent Beam Tests						
Distilled water	0/3**	-	NF1700***	0/3	-	NF1700
Tap water	0/3	-	↓	0/3	-	↓
3% NaCl sol.	0/3	-	↓	0/3	-	↓
0.25% Sodium dichromate	0/3	-	↓	0/3	-	↓
Soluble oil sol.	0/2	-	↓	0/3	-	↓
Cosmoline	0/3	-	↓	0/3	-	↓
High-humidity atmosphere	0/3****	-	↓	0/3	-	↓
Air	0/3	-	NF1700	0/3	-	NF1700
Solid propellant	0/0	-	-	0/0	-	-
Sea-coast exposure	0/0	-	-	0/0	-	-
U-Bend Tests						
High-humidity atmosphere	0/3	-	NF600	0/3	-	NF600
Trichloroethylene	0/0	-	-	0/0	-	-
Sea-coast exposure	0/0	-	-	0/0	-	-
Center-Notch Tests						
Distilled Water	0/2	-	NF100	0/2	-	NF100
3% NaCl sol.	0/2	-	↓	0/2	-	↓
0.25% Sodium dichromate	0/2	-	↓	0/2	-	↓
Soluble oil sol. (4%)	0/1	-	NF100	0/1	-	NF100

^{*} Refers to code letter in Master Schedule, Table 1.

^{**} Indicates no failures of three samples exposed.

^{***} Indicates no failures in 1700 hours exposure.

^{****} Indicates testing not started.

TABLE 5

STRESS-CORROSION CRACKING OF MARAGING STEELS IN VARIOUS ENVIRONMENTS

Test Environments	20% - Nickel Maraging Steel				18% - Nickel Maraging Steel							
	Material Condition H-1*	Material Condition H-3	Material Condition I-1	Material Condition I-3	Material Condition H-1*	Material Condition H-3	Material Condition I-1	Material Condition I-3				
	Failed/ Tested	Failure Time, hours	Failed/ Tested	Failure Time, hours	Failed/ Tested	Failure Time, hours	Failed/ Tested	Failure Time, hours				
Bent Beam Tests												
Aerated Distilled Water	3/3**	mean 11 range 10.2 - 18	1/3	1284	mean 1284 range 1284 - NF1650	3/3	mean 34.5 range 20.5 - 46.5	4/4	625	440 - 988		
Aerated Tap Water	0/3	-	NF1650***	1/3	1510	NF1650	2/3	350	325 - NF1550	0/3	-	NF1550
Aerated 3% NaCl Solution	3/3	7.3	6 - 8.5	0/3	-	NF1650	3/3	51.5	19 - 100	2/3	1290	1000 - NF1550
Aerated 0.25% Sodium Dichromate	1/3	1	1 - NF1650	0/3	-	NF1650	3/3	117	100 - 150	0/3	-	NF1550
4% Soluble Oil Solution	0/3	-	NF1650	0/3	-	NF1650	3/3	417	400 - 450	0/3	-	NF1550
Cosmoline Immersion	0/3	-	NF1650	0/3	-	NF1650	0/3	-	NF1550	0/3	-	NF1550
140°F Moisture-Saturated Air	3/3	100	22 - 174	2/3	1200	1080 - NF1650	3/3	21	20.5 - 21.5	3/3	260	245 - 290
75°F Air, 40% Humidity	0/3	-	NF1650	0/3	-	NF1650	0/3	-	NF1550	0/3	-	NF1550
Solid Propellant	0/0****	-	-	0/0	-	-	0/0	-	-	0/0	-	-
Seacoast Exposure	0/0	-	-	0/0	-	-	0/0	-	-	0/0	-	-
Center-Notch Tests												
Distilled Water	3/3	5.1	4.6 - 6.6	1/3	120.9	120.9 - NF300	3/3	85.3	83.1 - 87.0	2/2	13.2	12.6 - 13.8
3% NaCl Solution	2/2	7.2	6.6 - 7.8	2/2	40.2	34.4 - 46	2/2	20.6	18.0 - 23.2	2/2	5.9	5.0 - 6.9
0.25% Sodium Dichromate	0/2	-	NF200	0/2	-	NF100	1/2	67.9	67.9 - NF200	1/1	33.2	-
4% Soluble Oil Solution	0/1	-	NF200	0/1	-	NF100	0/1	-	NF150	0/0	-	-
Air	0/0	-	-	0/0	-	-	0/0	-	-	0/0	-	-

* Refers to material code letter of Table 1, Master Schedule.

** Indicates three failures out of three samples exposed.

*** Indicates no failures in 1270 hours of testing.

**** Indicates testing not started.

Table 5

TABLE 6

EVALUATION OF PROTECTIVE COATINGS ON H-11 JET
(FOR PREVENTING STRESS-CORROSION CRACKING)

Surface Condition	Coating	140°F Moisture-Saturated Air			Aerated 3% NaCl Solution		
		Failed/Tested	Failure Times, hours	Remarks	Failed/Tested	Failure Times, hours	Remarks
Surface Ground or Sanded	None	2/2*	64	48 - 70	4/4	1.6	0.3 - 2.5
	Polyurethane	6/6	3300	2550 - 5500	3/3	140	144 - 168
	Inhibited Epoxy 454-1-1	3/3	2720	2550 - 2850	0/0	-	-
	Inhibited Epoxy 463-1-1	3/3	655	400 - 970	0/3	-	MF1600**
	Inhibited Epoxy 463-4-8	3/3	845	289 - 1512	3/3	550	525 - 578
	Epoxy 463-1-1 over 454-1-1	4/4	4000	3550 - 4950	0/4	-	MF5350
	Zinc Silicate, Type 4	2/2	422	147 - 696	2/2	1.2	0.3 - 1.6
	80% Aluminum Epoxy	2/2	30	16 - 45	2/2	100	100 - 100
	70% Titanium Epoxy	2/2	195	136 - 256	2/2	150	140 - 160
	None	1/1	26.5	36.5	2/2	18.5	14 - 23
Sand Blasted	Pure Vinyl	0/2	-	VF200	0/2	-	MF200
	Zinc Silicate Type 4	0/2	-	VF200	2/2	14	10 - 18
	Zinc Silicate Type 4D with Cover Coat	0/2	-	VF200	2/2	76.7	1.5 - 152.5
	Inorganic Zinc Type 11	2/2	821	723 - 819	2/2	687	672 - 702
	Epoxy 188 over Inorganic Zinc Type 11	0/2	-	MF850	2/2	54	42 - 56
	Organic Zinc XI-4-245	2/2	766	742 - 790	2/2	214	27 - 100
	Modified Vinyl System	0/0***	-	-	0/0	-	-

* Indicates two failures out of two samples, exposed.

** Indicates no failures in 1600 hours exposure.

*** Indicates test not started.

Table 6

Best Available Copy